Understanding a ZnO nanorods fabrication process: a supersaturated design approach

Navin N. Acharya

Harold and Inge Marcus Department of Industrial and
Manufacturing Engineering,
The Pennsylvania State University,
310 Leonhard Building,
University Park, PA 16802, USA
E-mail: navin.acharya@gmail.com

Dennis K.J. Lin*

Supply Chain and Information Systems,
The Pennsylvania State University,
483 Business Building,
University Park, PA 16802, USA
Fax: +1-814-863-7067
E-mail: DKL5@psu.edu
*Corresponding author

Abstract: Nanomanufacturing promises to increase quality, productivity and efficiency of existing technologies and has the potential to accelerate commercialisation of products and benefit various industries. As this technology is still in its discovery stage, there is a tremendous amount of experimentation occurring every day. Often in a nanomanufacturing setup, a large number of factors can be listed as possible sources of effects and among those, only a few are actually significant. A problem frequently encountered in nanomanufacturing is how to reduce the total number of experiments while estimating a large number of effects. Realising this challenge and the growing application of statistical techniques to discover relationships at the nanoscale, we advocate the use of supersaturated designs to nanomanufacturing settings. Here, we develop a supersaturated design, which is effective in identifying significant effects with minimal experimental runs for a process for the fabrication of ZnO nanorods, and discuss the analysis techniques.

Keywords: design of experiments; nanomanufacturing scaling-up; screening experiment; supersaturated design; variable selection; ZnO nanorods.

Reference to this paper should be made as follows: Acharya, N.N. and Lin, D.K.J. (2008) 'Understanding a ZnO nanorods fabrication process: a supersaturated design approach', *Int. J. Nanomanufacturing*, Vol. 2, No. 4, pp.319–330.

Biographical notes: Navin N. Acharya is a Member of the American Society for Quality (ASQ) and of the Institute of Industrial Engineers (IIE). He has received his PhD in Industrial Engineering from The Pennsylvania State University in 2006.

Dennis K.J. Lin is a University Distinguished Professor of Supply Chain Management and Statistics at The Pennsylvania State University. His research interests are quality engineering, industrial statistics, data mining and response surface. He has published over 120 papers in a wide variety of journals. He is an Elected Fellow of the American Statistical Association (ASA), an Elected Member of International Statistical Institute (ISI), an Elected Fellow of American Society of Quality (ASQ), a Lifetime Member of International Chinese Statistical Association (ICSA) and a Fellow of the Royal Statistical Society (RSS). He is also the recipient of the 2004 Faculty Scholar Medal Award at Penn State University.

1 Introduction

Scientific properties of the atoms combined to create a structure are still greatly unknown. As every atom being considered can potentially have a significant impact on the nanostructure, high-precision manufacturing techniques need to be considered at the nanoscale. Although the bottom-up approach has been applied with reasonable success, its application has been limited due to a host of reasons. A workshop conducted by the National Science Foundation (NSF) (see Busnaina et al., 2003) recognises that one of the foremost challenges that nanomanufacturing faces is that of scaling-up. Scaling-up can be defined as the translation of the research discoveries in the laboratory to commercially available products. In order to effectively scale-up nanotechnology to achieve mass production, several key issues need to be addressed. Some of these key issues have been recognised as low reliability and yield for nanoscale devices, repeatability and reproducibility in yielding a particular product, and lack of control of the nanomanufacturing processes.

One possible way of overcoming some of the limitations of nanomanufacturing, especially that of scaling-up and potentially gaining a comprehensive understanding of the properties and characteristics at the nanoscale is through the effective use of designed experiments. In particular, Nembhard et al. (2005) demonstrate the successful use of designed experiments in understanding the characteristics of C-6 alcohol molecules for gas-phase lubrication. The authors call for new constructs of designs that can be applied to a wide genre of nanomanufacturing processes.

One such common occurrence is when the number of parameters to be estimated in this study is extensive and the resources limited. In early stages of industrial experimentation, one often has a large set of candidate factors believed to have possible significant influence on the response of interest, although it is reasonable to assume that only a small fractional are influential, a condition known as effect sparsity. In this paper, one such example (fabrication of ZnO nanorods) is studied where the experimenter has to deal with many process variable and limited experimental runs. Design of an experiment with the property that the number of potential effects exceeds the number of experimental runs (observations) is called a supersaturated design. These designs are useful as screening tools. Supersaturated designs, proposed by Lin (1993), can be extremely helpful in discovering complex relationships within a large set of experimental variables, given limited resources. Detailed study and sound control over the process will aid in practical and large-scale implementation of the nanostructure(s) being developed. The supersaturated design would be extremely helpful in understanding the above fabrication process while utilising minimal resources.

Although the use of structured experimental designs is not widespread at the nanoscale, many researchers have employed various DOE techniques. These designs are used to investigate input variables of a particular process, or obtain a model to predict conditions that would yield desired results or optimise process conditions to successfully achieve scaling-up. A brief survey of the literature for the past few years that incorporate some form of experimental design is given below.

Improvements in the rheological behaviour of the nanosilica composite no-flow underfill were achieved by Sun et al. (2004) by investigating the experimental conditions of the surface treatment using silane-coupling agents with the help of a designed experiment. Although no details on the choice of design were mentioned, it seems as though a fractional factorial design, with two baseline runs, was used for the four input variables. The significant main effects, along with an interaction term, were identified, and the input variables were set at corresponding operating conditions for further characterisation.

Another instance of the use of DOE is given in Kharbas et al. (2003). Two L16 Taguchi orthogonal array fractional factorial designs were executed, one each for neat resin and nanocomposite. Five process parameters were varied over four levels each in the designs. Fractionation was chosen to ensure than maximum number of factors of interest could be identified. Thus, through DOE, optimal process conditions that would result in desirable cell size and density, thus better mechanical properties, were attained.

A robustness study was also conducted by Dewey et al. (2000) for modelling Micro-Electro-Mechanical Systems (MEMS) to minimise effects of device parametric variability on overall performance. Taguchi designs were used effectively in identifying factors most influential to the process output and determining the settings of the parameters that yield both an acceptable performance metric and minimise variations. Along similar lines, Ren et al. (2001) propose a three-step technique for quality optimisation of MEMS devices using Taguchi methods. Taguchi designs were also employed by Jackson et al. (2005) to optimise a Time-Modulated Chemical Vapour Deposition (TMCVD) process. Time, money and effort were saved by adopting the experimental design approach in the deposition process of nanocrystalline diamond coating.

A common trend seen is the application of structured experimental designs in investigating the behaviour of processed being considered. Some designs are more advanced than others are. Furthermore, in many other studies, there is an indication of some structure in the manner in which experiments were conducted, but no details mentioned. For instance, Grisolia et al. (2005) studied two annealing conditions by varying process parameters such as time, temperature and ambient atmosphere. The experimental details hint the use of a multiway ANOVA, but no discussion is provided. From past literature, it is noticed that although DOE has been employed to study some of the processes at nanoscale, advanced techniques have been overlooked for the sake of simplicity in experimentation. Specifically, in studies involving many variables and fewer runs, supersaturated designs are not understood well enough to be employed. For more such examples, see Jancar and Suvorov (2006), Zhang et al. (2005) and Boal et al. (2006).

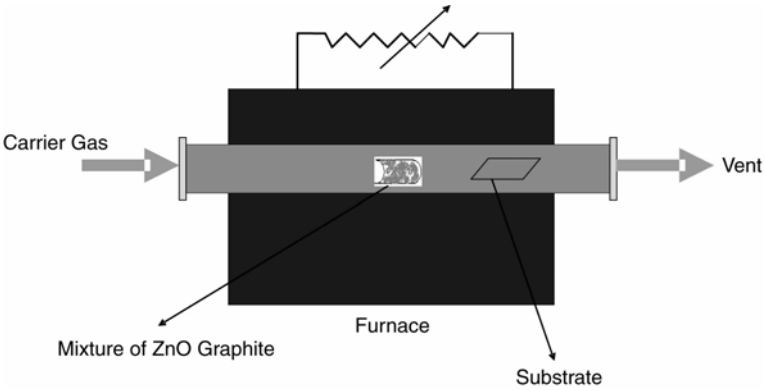
The rest of this paper is organised as follows. Section 2 explains the nanomanufacturing process for the fabrication of nanorods. As traditional statistical designs fail to accommodate the needs and constraints of this process, a new class of

supersaturated designs is applied to the process. The need for these types of designs, their properties, and analysis techniques are explained in Section 3. The detailed application of a supersaturated design to the process is presented in Section 4. Finally, Section 5 draws conclusions and a general discussion is provided as well.

2 Nanomanufacturing setup

The setting for the fabrication of zinc oxide (ZnO) provided motivation to study the potential of supersaturated designs in its application to nanomanufacturing settings. ZnO can be used for a wide variety of applications such as short-wavelength light-emitting, UV lasing and nanoresonators. Kumar et al. (2005, 2006) study the synthesis and assembly of these ZnO structures using gas phase synthetic methods. They state that ZnO generally exhibits a variety of structures and these differences in morphology can be attributed to various settings of the process variables. Figure 1 represents a schematic of the experimental setup used by Kumar et al. (2005).

Figure 1 Schematic for the fabrication of ZnO nanorods



In this process, a catalyst is grown in a predefined chemical growth medium and a substrate prepared (usually diluted in de-ionized water). To pattern substrates with catalysts at predetermined locations, periodic patterns are constructed. For homogenous catalyst deposition, a bacterial solution is deposited on the substrate in the presence of a magnetic field. A mixture of ZnO powder and graphite is heated at a specified temperature, for a specified time, and under a specified gas flow rate so that the ZnO structures can be grown on the substrate. The mixture is placed in the centre of a furnace and the substrate is placed at a specified distance away in the downstream direction from the mixture. The optical quality (morphology) of the ZnO nanorods is studied using an Atomic Force Microscope (AFM) and the response to be measured is the surface roughness of the nanorods.

The overall objective is to grow ZnO nanorods with consistent measurements of surface roughness. Increased understanding of the process variables will lead to greater control of the dimension, composition, position and orientation of the NRs, which will eventually lead to facilitation of photonic-based application of NRs as optoelectronic devices and chemical/biological sensors. True potential of ZnO nanorods can be realised only when a thorough understanding of the process conditions, along with control of the

morphology of the nanostructure is achieved. Although significant progress in terms of the ability to fabricate nanorods using biocatalysts has been made, the interdependencies between the process variables might reveal important details that may make the fabrication process more reliable, robust and reproducible. The first step in doing so is to identify – in consultation with the experimenter – the factors that might affect the optical quality of the nanorods. These factors are listed in Table 1 along with the levels at which they are to be tested for significance.

Table 1 List of process variables identified

Label	*Variable*	*Low (–) level*	*High (+) level*
A	Substrate	Silicon	Gold
B	Carrier gas	Argon	Nitrogen
C	Process temperature	500ºC	1000ºC
D	Carrier gas flow rate	100 sccm	150 sccm
E	Synthesis time	1 hr`	3 hr
F	Catalyst	Magnetospirillum magnetotacticum (MS-1)	Cobalt
G	Distance between powder and substrate	5"	10"
H	Optical density of bacterial solution	0.12	0.15
J	Time for depositing bacterial solution on substrate	30 min	1 hr

Currently, the researchers synthesise the nanorods based on the settings corresponding to the 'low' levels in Table 1. The researchers would like to study the significance of the above factors on the optical quality of the nanorods by including another level (given by 'high' in Table 1) for each process variable. An appropriate design that systematically studies the above experimental setup was developed and is presented in the next two sections.

3 Supersaturated designs: the need for them and their characteristics

As in any manufacturing process, there are constraints imposed either due to limited resources or the experimental setup itself. In the process for the fabrication of ZnO nanorods, one of the constraints imposed on the above process is the fact that it takes about Six hours every time the process temperature needs to be adjusted to a new level. If the time required for depositing the solution on the substrate (factor *J*) and the synthesis time (factor *E*) are added to the time for temperature change, each individual run (defined as the creation of nanorods using any one particular setting in Table 1) will take at least 7.5 hr. Finally, a maximum of 12 experimental runs were deemed feasible for initial experimentation. Hence, in order to test all the nine process variables and the two-way interactions between them, a regular factorial design (full or fractional) would require an unacceptable amount of time. In addition, the researchers believe that only a few of the factors listed might actually be responsible in controlling the roughness of the nanorods.

This principle of 'effects sparsity' was explained by Lin (1993) who proposed supersaturated designs to reduce the total number of runs. A supersaturated design is basically a fractional factorial design in which the number of experimental runs is less than the number of parameters to be estimated. These designs are used as screening tools, as the paring of candidate factors is performed in a cost-efficient manner. In order to obtain an unbiased estimate of the main effect of each factor, the number of experimental runs must exceed (or at least equal to) the number of factors plus one (for estimating the overall grand average). When these two are equal, the design is called a saturated design and is the minimum effort required to estimate all main effects. The standard advice given to researchers conducting a process similar to the one described in the previous section is to use the saturated design, which is 'optimal' based on certain theoretical optimality criteria. However, the non-significant effects are not of interest. Estimating all main effects may be wasteful if the goal is simply to detect those few active factors. If the number of active factors is indeed small, the use of a slightly biased estimate will still allow one to accomplish the identification of active factors but significantly reduce the amount of experimental work (time and/or resources). This is particularly important in situations where the cost of an individual experimental run is expensive or simply too time consuming.

A new class of supersaturated designs is defined by Lin (1993) as a factorial design with n observations and k factors, such that $k > n - 1$. These designs can easily be constructed using half fractions of Hadamard matrices. These designs can examine $k = N - 2$ factors with $n = N/2$ runs, where N is the order of the Hadamard matrix used. The Plackett and Burman (1946) designs, which can be viewed as a special class of the Hadamard matrices, are used to illustrate the basic construction method.

Table 1 A supersaturated design derived from the hadamard design of order 12

R	#	*1*	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*
	1	+	+	–	+	+	+	–	–	–	+	–
1	2	+	–	+	+	+	–	–	–	+	–	+
2	3	–	+	+	+	–	–	–	+	–	+	+
	4	+	+	+	–	–	–	+	–	+	+	–
3	5	+	+	–	–	–	+	–	+	+	–	+
4	6	+	–	–	–	+	–	+	+	–	+	+
5	7	–	–	–	+	–	+	+	–	+	+	+
	8	–	–	+	–	+	+	–	+	+	+	–
	9	–	+	–	+	+	–	+	+	+	–	–
	10	+	–	+	+	–	+	+	+	–	–	–
6	11	–	+	+	–	+	+	+	–	–	–	+
	12	–	–	–	–	–	–	–	–	–	–	–

Table 2 shows the original 12-run Plackett and Burman design. Take column 11 as a branching column, meaning that the 12 runs are divided into two groups of 6 runs each, in accord with the sign in column 11. Then the runs (rows) can be split into two groups: Group I with the sign of +1 in column 11 (rows 2, 3, 5, 6, 7 and 11), and Group II with

the sign of –1 in column 11 (rows 1, 4, 8, 9, 10 and 12). Deleting column 11 from Group I causes columns 1 – 10 to form a supersaturated design to examine $N - 2 = 10$ factors in $N/2 = 6$ runs (runs 1 – 6 as indicated in Table 3). It can be shown that if Group II is used, the resulting supersaturated design is an equivalent one. In general, a Plackett and Burman design matrix can be split into two half-fractions according to a specific branching column whose signs equal +1 and –1. Specifically, take only the rows which have +1 in the branching column. Then the $N - 2$ columns other than the branching column will form a supersaturated design for $N - 2$ factors in $N/2$ runs. These designs have shown to be optimal among all other supersaturated designs. For more properties of supersaturated designs, refer to Lin (1995, 2003) and Deng et al. (1999).

Table 2 The resulting supersaturated design for $(n, k) = (6, 10)$

No	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}
1	+	–	+	+	+	–	–	–	+	–
2	–	+	+	+	–	–	–	+	–	+
3	+	+	–	–	–	+	–	+	+	–
4	+	–	–	–	+	–	+	+	–	+
5	–	–	–	+	–	+	+	–	+	+
6	–	+	+	–	+	+	+	–	–	–

Finally, the interaction columns of Hadamard matrices are only partially confounded with other main effect columns (Lin and Draper, 1992, 1993). Wu (1993) makes use of such a property and proposes a supersaturated design that consists of all main-effect and two-factor interaction columns from any given Hadamard matrix of order N. The resulting design has N runs and can accommodate up to $N(N - 1)/2$ factors, namely, $N - 1$ columns from main effects and $(N - 1)(N - 2)/2$ columns from interaction effects. When there are $k < N(N - 1)/2$ factors to be studied, choosing columns becomes an important issue to be addressed. Provided the interactions are not fully confounded with the main effects, Lin (1998) extends this idea to analyse interaction effects for first-order designs.

4 Application of supersaturated designs to the nanomanufacturing setup

For the process of fabricating ZnO nanorods, we have identified nine process variables (factors) that can potentially have an effect on the morphology of the fabricated nanorods. In addition to these process variables, the two-way interactions between factors are also considered to be of interest by the experimenters. Thus, there are ${}^{9}C_{2} = 36$ two-way interaction effects to be examined. In summary, there are 45 parameter effects (9 main and 36 two-way) to be examined. Due to the experimental constraints imposed by the nature of the process, a maximum of 12 experimental runs can be performed. Given these limitations, the objective is to design an experiment to identify the most significant process parameters in terms of impact on the response. For the given setup, a supersaturated design is the most appropriate as the number of

parameters to be estimated (45) far exceed the number of experimental runs (12). Traditional fractional factorial designs require a high degree of fractionation in order to limit the number of experimental runs to 12. Due to the heavy fractionation, it is impossible to differential the effects of two-way interactions with main effects.

From the previous sections, $n = 12$, $k = 45$. As $k > n - 1$, a supersaturated design is constructed for the process using the method described in Section 3 using a Hadamard matrix of order N, where $N = 12$. The design was constructed from the 12 run Plackett and Burman design given in Table 2. This design is capable of accommodating at most 11 factors. As we have nine process variables of interest, we constructed a supersaturated design using the first nine columns of Table 2. Column 11 was used as a branching column to generate Group 1 (# 2, 3, 5, 6, 7 and 11 from Table 2) and Group 2 (# 1, 4, 8, 9, 10 and 12 from Table 2). These groups caused columns 1–9 to form a supersaturated design. The resulting supersaturated design for the fabrication of ZnO nanorods is given below in Table 4. Columns $A - J$ represent the main effects of the factors. Columns corresponding to the two-way interaction terms were created by multiplying the two appropriate columns. For example, column for parameter AB can be created by multiplying columns A and B. For the sake of brevity, only the first nine of the total 45 parameters of interest are shown in Table 4. From Section 3, it is clear that the two-way interaction effects are only partially confounded with other main effects, and hence estimable.

Table 3 Supersaturated design for the fabrication process

Run									
Group 1	*A*	*B*	*C*	*D*	*E*	*F*	*G*	*H*	*J*
1	+	–	+	+	+	–	–	–	+
2	–	+	+	+	–	–	–	+	–
3	+	+	–	–	–	+	–	+	+
4	+	–	–	–	+	–	+	+	–
5	–	–	–	+	–	+	+	–	+
6	–	+	+	–	+	+	+	–	–
Group 2									
7	+	+	–	+	+	+	–	–	–
8	+	+	+	–	–	–	+	–	+
9	–	–	+	–	+	+	–	+	+
10	–	+	–	+	+	–	+	+	+
11	+	–	+	+	–	+	+	+	–
12	–	–	–	–	–	–	–	–	–

The execution of the above design is as important as its construction. If the design is not executed in the manner in which it is indented, the results from the analysis can be seriously misleading. Supersaturated designs are executed in a fashion similar to a randomised complete block factorial design. In other words, within a group, after each experimental run, the settings of all process variables are reset. Also, the order of the experimental runs itself are randomised. The best way of doing so is to first choose any of the two groups randomly. Within a chosen group, have 6 pieces of paper, each having

a number from 1 to 6. Each numbered paper will then correspond to a run in Table 4, which will in turn correspond to a particular combination of process variables $A - J$. For instance, run 7 corresponds to studying the roughness of the nanorods by maintain the settings of the process variables as follows.

- Gold substrate ('+' level of factor *A*)
- Nitrogen carrier gas ('+' level of factor *B*)
- Process temperature at 500°C ('–' level of factor *C*)
- 150 sccm as the carrier gas flow rate ('+' level of factor *D*)
- 3 hr of synthesis time ('+' level of factor *E*)
- Cobalt as the catalyst ('+' level of factor *F*)
- Place the powder 5 inches from the substrate ('–' level of factor *G*)
- 0.12 as the optical density of the bacterial solution ('–' level of factor *H*)
- 30 min for depositing bacterial solution on the substrate ('–' level of factor *J*).

A random selection of a numbered paper will dictate the settings of the process variables to be used. At the end of each experimental run, the morphology of the fabricated nanorods is examined and responses (roughness levels) recorded. Six observations will be recorded in the first chosen group. The above process is carried out for the other group as well. Thus, in all 12 observations are recorded. The next step is to analyse the data in order to make recommendations on the significant process variables. These significant process variables can be used, with great benefit, for future follow-up experimentation.

The final step of the initial screening experiment is to analyse the data once all the observations are recorded. Plackett and Burman designs are traditionally known as main-effect designs because, if all the interactions are ignored, they can be used to estimate all the main effects (Lin, 2003). The main-effects model can be analysed in several ways, the most common being the use of a normal probability plot (Montgomery, 2005). If an effect is at least three times larger than the overall random error standard deviation, such an effect can virtually always be detected. In the presence of interaction parameters, a regular stepwise regression analysis (Kutner et al., 2005; Lin, 1995) is best suited. This procedure starts with no variable in the model and first selects the $\boldsymbol{x_i}$ that has the highest correlation with $\boldsymbol{y}$ (where $\boldsymbol{x_i}$ is a column representing effect i and $\boldsymbol{y}$ is the response column). Subsequent selections are based on partial correlations, given the variables already selected. The stepwise selection procedure is available in most statistical software packages. This is a powerful and convenient method to identify significant effects. Most programmes allow the analyst to select the criterion to enter a new variable and also to remove one.

In general, the following steps can be applied for the analysis of a supersaturated design with interaction parameters to be estimated.

- Generate all interaction columns, and combine them with the main-effect columns. We now have $k(k + 1)/2$ columns.
- Analyse these $k(k + 1)/2$ columns with n experimental runs using a regular stepwise regression technique for model building.

For the sake of illustration, assume that the experiment for understanding ZnO fabrication process was carried out as per the instructions given above and the responses recorded for each of the 12 runs. Using standard statistical packages such as SAS®, a stepwise regression analysis could then be conducted in order to understand the relationships between factors. If, for instance, main effect *A* (substrate type) and interaction effect *BD* (carrier gas and carrier gas flow rate) are found to be the most significant, the next step is to observe the main effects plot and interaction plot for the significant effects identified. By studying these plots, the researcher can then choose optimal settings of the significant effects such that the variability in the response is minimal (thus obtaining consistent measurements of surface roughness). For more information on main effects plots and interaction plots, please refer to Montgomery (2005).

Thus, we constructed a supersaturated design to study the impact of various process variables on the optical quality of the nanorods in a ZnO fabrication process. Data can be recorded by following the above instructions provided to the experimenter, and analysed using stepwise regression, which is available in most commercial statistical software packages.

5 Conclusion and discussion

Bottom-up approaches for fabricating nanostructures provides a crucial building block to create devices and structures atom-by-atom or molecule-by-molecule. Currently, processes employing bottom-up approach are been investigated by researchers and are considered as the state-of-the-art in fabrication of structures at the nanoscale. In order to tap into the full potential of nanomanufacturing and discover relationships between the process variables that would lead to better control and understanding of processes, we propose the use of supersaturated designs. Supersaturated designs are very useful in early stages of the experimental investigation of complicated systems and processes involving many factors. They are not used for a terminal experiment. Knowledge of the confounding patterns makes possible the interpretation of the results and provides the understanding of how to plan the follow-up experiments.

Since the study of *random balanced design* in late 1950s (Satterthwaite, 1959), the work of supersaturated design has been dormant until the appearance of Lin (1993). The related work about supersaturated design is now much more mature, from better design construction, to clearer understanding on design capability, to more reliable data analysis methods. In today's nanotechnology era, we face more and more variables/parameters, the use of supersaturated design is considered to be a relatively novel endeavour.

Using supersaturated designs involves more risk than using designs with more runs. However, their use is far superior to other experimentation approaches such as subjective selection of factors or changing factors one-at-a-time. The latter can be shown to have irresolvable confounding patterns, though such confounding patterns are important for data analysis and follow-up experiments. The success of a supersaturated design depends heavily on the 'effect sparsity' assumption. Supersaturated designs are most suitable for cases where the number of parameters is much greater than the number of runs. In fact, greater the number of parameters to be estimated, greater is the capability of supersaturated designs.

More and more researchers are benefiting from using computational power to construct designs for specific needs. Lin (1995) introduced the first computer algorithm to construct supersaturated designs. Such an algorithm seems to perform well for constructing supersaturated designs by various criteria. Commercial statistical software for constructing supersaturated designs is also available; see for example, JMP®.

References

Boal, A.K., Bachand, G.D., Rivera, S.B. and Bunker, B.C. (2006) 'Interactions between cargo-carrying biomolecular shuttles', *Nanotechnology*, Vol. 17, pp.349–354.

Busnaina, A., Barry, C. and Miller, G. (2003) *NSF Workshop on Three Dimensional Nanomanufacturing: Partnering with Industry; Conclusions and Report*, Available at: http://www.nano.neu.edu/nsf_workshop.html. Accessed on 21 August 2005.

Deng, L.Y., Lin, D.K.J. and Wang, J.N. (1999) 'On resolution rank criterion for supersaturated design', *Statistica Sinica*, Vol. 9, pp.605–610.

Dewey, A., Ren, H. and Zhang, T. (2000) 'Behavioral modeling of microelectromechanical systems (MEMS) with statistical performance-variability reduction and sensitivity analysis', *IEEE Transactions on Circuits and Systems II: Analog and Digital Signal Processing*, Vol. 47, No. 2, pp.105–113.

Grisolia, J., Shalchain, M., Assayag, G.B., Coffin, H., Bonafos, C., Schamm, S., Atarodi, S.M. and Claverie, A. (2005) 'The effects of oxidation conditions on structural and electric properties of silicon nanoparticles obtained by ultra-low-energy ion implantation', *Nanotechnology*, Vol. 16, pp.2987–2992.

Jackson, M.J., Robinson, G.M., Ahmed, W., Sein, H. and Jones, A.N. (2005) 'Time-modulated chemical vapor deposition of diamond films', *Journal of Materials Engineering and Performance*, Vol. 14, No. 2, pp.163–172.

Jancar, B. and Suvorov, D. (2006) 'The influence of hydrothermal-reaction parameters on the formation of chrysotile nanotubes', *Nanotechnology*, Vol. 17, pp.25–29.

Kharbas, H., Nelson, P., Yuan, M., Gong, S. and Turng, L. (2003) 'Effects of nano-fillers and process conditions on the microstructure and mechanical properties of microcellular injection molded polyamide nanocomposites', *Polymer Composites*, Vol. 24, No. 6, pp.655–671.

Kumar, N., Dorfman, A. and Hahm, J. (2005) 'Fabrication of optically enhanced zno nanorods and microrods using novel biocatclysts', *Journal of Nanoscience and Nanotechnology*, Vol. 5, pp.1–4.

Kumar, N., Dorfman, A. and Hahm, J. (2006) 'Ultrasensitive DNA sequence detection using nanoscale zno sensor arrays', *Nanotechnology*, Vol. 17, pp.2875–2881.

Kutner, M.H., Nachtsheim, C.J., Neter, J. and Li, W. (2005) *Applied Statistical Linear Models*, 5th edition, New York, NY: McGraw-Hill Irwin.

Lin, D.K.J. (1993) 'A new class of supersaturated designs', *Technometrics*, Vol. 35, No. 1, pp.28–31.

Lin, D.K.J. (1995) 'Generating systematic supersaturated designs', *Technometrics*, Vol. 37, pp.213–225.

Lin, D.K.J. (1998) 'Spotlight interaction effects in main-effect designs', *Quality Engineering*, Vol. 11, pp.133–139.

Lin, D.K.J. (2003) 'Industrial experimentation for screening', in C.R. Rao and R. Khattree (Eds). *Handbook of Statistics in Industry*, Chapter 2, Vol. 22, pp.33–73, North Holland, New York.

Lin, D.K.J. and Draper, N.R. (1992) 'Projection properties of Plackett and Burman designs', *Technometrics*, Vol. 34, pp.423–428.

Lin, D.K.J. and Draper, N.R. (1993) 'Generating alias relationships for two-level Plackett and Burman designs', *Computational Statistics and Data Analysis*, Vol. 15, pp.147–157.

Montgomery, D.C. (2005) *Design and Analysis of Experiments*, 6th edition, New York, NY: John Wiley and Sons Inc.

Nembhard, H.B., Acharya, N.N., Aktan, M. and Kim, S. (2005) *Design Issues and Analysis of Experiments in Nanomanufacturing*, Chapter 20, CRC Press Handbook of Industrial and Systems Engineering.

Plackett, R.L. and Burman, J.P. (1946) 'The design of optimum multifactorial experiments', *Biometrika*, Vol. 33, pp.303–325.

Ren, H., Jog, A. and Fair, R.B. (2001) 'Statistical optimal design of microelectromechanical systems (MEMS)', *International Conference on Modeling and Simulation of Microsystems MSM*, San Diego, CA, 19–21 March.

Satterthwaite, F. (1959) 'Random balance experimentation (with discussion)', *Technometrics*, Vol. 1, pp.111–137.

Sun, Y., Zhang, Z. and Wong, C.P. (2004) 'Fundamental research on surface modification of nano-size silica for underfill applications', *9th International Symposium on Advanced Packaging Materials*, pp.132–138.

Wu, C.F.J. (1993) 'Construction of supersaturated designs through partially aliased interactions', *Biometrika*, Vol. 80, pp.661–669.

Zhang, T., Xu, Z., Tao, D.L., Teng, F., Li, F.S., Zheng, M.J. and Xu, X.R. (2005) 'The influence of ZnO nanorods on the Poly-(3, 4-Ethylenedioxythiophene): Poly-(Styrenesulphonic Acid) Buffer Layer in a Polymer Light-Emitting Diode', *Nanotechnology*, Vol. 16, pp.2861–2864.